#
11
05-01-02

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of
Takayuki WATANABE et al.
Serial No. 09/492,137
Filed: January 27, 2000
For: EDIBLE POWDER MATERIAL HAVING EXCELLENT SHELF STABILITY

Docket No. 00202/K-5(Hase) F99023
Group Art Unit 1761
Examiner H. Mai

DECLARATION UNDER RULE 1.132

Honorable Commissioner of
Patent and Trademarks
Washington, D.C.

Sir:

I, Hisashi Suzuki, hereby declare as follows:

That I graduated, in March 1991, at Obihiro University of Agricultural Science and Veterinary Medicine, Department of Agricultural Chemistry;

That, in April 1991, I joined T. Hasegawa Co., Ltd., and was assigned to Technical Research Center, where I have since mainly engaged in the research and development of powdered flavourings up to the present;

That I am one of the co-inventors of U.S. Application Serial No. 09/492,137;

That the following experiments were carried out by myself, or under my supervision and control.

Experiment 1A (according to the present invention)

In 100 g of water, there were added to and dissolved 20 g of water-soluble hemicellulose (SOYAFIVE-SLA200 manufactured by Fuji Oil Co., Ltd., with an average molecular weight of about 200,000) and 60 g of trehalose. The resultant solution was sterilized by heating at 85-90℃ for 15 minutes. After the solution was cooled to 60℃, 20 g of edible fats and oils which contained β-carotene in a concentration of 5 % was added to the solution and mixed. The resulting mixture was emulsified with TK-Homomixer (trade name of a mixer manufactured by Tokushu Kika Kogyo Co., Ltd.). With use of a Mobile Minor type spray dryer (manufactured by Niro Inc.), the resultant emulsion was spray-dried at an inlet temperature of 160℃ and an outlet temperature of 80℃ to give 92 g of β-carotene-containing powders (Inventive product 1A).

Experiment 1B (Comparative Example)

The operation of the above Experiment 1A was repeated except that 60 g of trehalose was replaced with 60 g of dextrin which was well known as excipient, and, thus, there was obtained 94 g of β-carotene-containing powders (Comparative product 1B).

Experiment 1C (Comparative Example)

The operation of the above Experiment 1A was repeated except that 20 g of water-soluble hemicellulose was replaced with 20 g of octenyl succinic acid-esterified starch which was well known as food emulsifier, and, thus, there was obtained 90 g of β-carotene-containing powders (Comparative product 1C).

[Storage Stability Test]

β-Carotene-containing powders which had been obtained in the

above-mentioned Experiments 1A to 1C were subjected to storage test as shown below, and were measured for the content of β-carotene with use of high-performance liquid chromatography. Results are shown in Table A. The retention of β-carotene in Table A is a relative percentage of β-carotene which remained after four-week storage, to the content of β-carotene (100 %) immediately after preparation.

Storage test method

① β-Carotene-containing powders were put into a low-density polyethylene bag, which was stored in the dark at 50℃ for four weeks.

② β-Carotene-containing powders were put into a high-density polyethylene bag, which was stored under fluorescent lamp illumination at 4,500 lx at a temperature of 25℃ for four weeks.

Table A: Retention of β-carotene

	After stored in the dark at 50℃ for 4 weeks	After stored under fluorescent lamp illumination at 4,500 lx at a temperature of 25℃ for four weeks
Inventive product 1A	98.7 %	97.6 %
Comparative product 1B	66.3 %	54.1 %
Comparative product 1C	43.3 %	35.2 %

As is seen in the above, β-carotene-containing powders exhibit no sufficient storage stability unless both water-soluble hemicellulose and trehalose are present.

Experiment 2A (according to the present invention)

In 100 g of water, there were added to and dissolved 20 g of water-soluble hemicellulose (SOYAFIVE-SLA200 manufactured by Fuji Oil Co., Ltd., with an average molecular weight of about 200,000) and 60 g of trehalose. The resultant solution was sterilized by heating at 85-90℃ for 15 minutes. After the solution was cooled to 60℃, 20 g of lomon flavor was added to the solution and mixed. The resulting mixture was emulsified with TK-Homomixer (trade name of a mixer manufactured by Tokushu Kika Kogyo Co., Ltd.). With use of a Mobile Minor type spray dryer (manufactured by Niro Inc.), the resultant emulsion was spray-dried at an inlet temperature of 160℃ and an outlet temperature of 80℃ to give 90 g of lemon powder perfume (Inventive product 2A).

Experiment 2B (Comparative Example)

The operation of the above Experiment 2A was repeated except that 60 g of trehalose was replaced with 60 g of dextrin which was well known as excipient, and, thus, there was obtained 91 g of lemon powder perfume (Comparative product 2B).

Experiment 2C (Comparative Example)

The operation of the above Experiment 2A was repeated except that 20 g of water-soluble hemicellulose was replaced with 20 g of octenyl succinic acid-esterified starch which was well known as food emulsifier, and, thus, there was obtained 90 g of lemon powder perfume (Comparative product 2C).

[Storage Stability Test]

According to the process below, tablets were prepared by adding 0.5 % of individual lemon powder perfumes which had been obtained in the

above-mentioned Experiments 2A, 2B and 2C. Thus prepared tablets were subjected to storage test as shown below, and were then organoleptically examined by expert panelists for fragrance and flavor. Results are shown in Table B.

Method for the preparation of tablets

(Formulation)

Raw materials	Amount used
1. Powder sugar	903 (g)
2. Lactose	30
3. Vitamin C	37
4. Citric acid powder	15
5. 1 % aqueous solution of gelatin	40
6. Sucrose fatty acid ester	10
7. Lemon powder perfume	5
Total	1,040
Dry weight	1,000

(Process)

(1) After powder materials 1. to 4. were mixed, material 5. was added, and the resultant mixture was agitated until it became homogeneous.

(2) The resulting blend was granulated to a size of less than 30 mesh.

(3) Thus obtained granules were dried at 45℃ for 60 minutes.

(4) Materials 6. and 7. were added to, and mixed with, the granules dried in (3).

(5) Thus obtained blend was tableted under the following conditions:

Weight: 1.8 g/tablet

Diameter: 2 cm

Pressure: 40 kg/cm²/tablet

Storage test method

① Tablets were put into a low-density polyethylene bag, which was stored in the dark at 50℃ for four weeks.

② Tablets were put into a high-density polyethylene bag, which was stored under fluorescent lamp illumination of 4,500 lx for two weeks.

As a control, tablets were put into an aluminum bag, which was stored at −18℃.

Table B: Results of Organoleptic Examination

	After storage at −18℃ (Control)	After stored in the dark at 50℃ for 4 weeks	After storage at 4,500 lx for two weeks
Inventive product 2A	10	9	8
Comparative product 2B	10	3	2
Comparative product 2C	10	2	2

The numbers in the above table show relative evaluation of organoleptic examination of each of the products in comparison with the evaluation after stored at −18℃ which is supposed to be 10. The smaller the numbers are, the more deteriorated are fragrance and flavor.

As is seen in the above, lemon powder perfume exhibits no sufficient storage stability unless both water-soluble hemicellulose and trehalose are present.

The undersigned declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application of any patent issuing thereon.

Signed this 25th day of April, 2002

Hisashi Suzuki.

Hisashi Suzuki